# ELECTRICAL CONDUCTIVITY MODELS OF DIE CONFIGURATION FOR POLYPROPYLENE-REINFORCED MILLED CARBON FIBRE

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**ABSTRACT:** Extrusion is one of the pre-mixing processes that produce a highly conductive polymer composite material when the appropriate die geometry is applied. However, the development of a conductivity model is still in the preliminary stage because the current model being used is unable to accurately predict the electrical conductivity. A generally effective media model and a modified fibre contact model were adapted in this study. To obtain a good agreement between the predicted model and the experimental data, the FCM model was modified by adapting the extrusion parameters, including the shear rate and rotational speed, into the equation. This was known as the MFCM with shear rate. This modified MFCM with shear rate showed a good correlation with the r-square of 0.99, as evidenced by the minimized gap between the predicted and experimental data. The MCF/PP composite produced using the rod die had the highest electrical conductivity of 3.7 S/cm as the fibre was aligned in the converging dies than in the sheet dies, which had an electrical conductivity of 1.3 S/cm.

**KEYWORDS**: *Extrusion; Electrical Conductivity; Carbon Fibre; Modelling*

## 1.0 INTRODUCTION

Manufacturing processes, such as injection moulding and compression moulding, are the primary methods used for producing highly conductive polymer composite (CPC) materials [1]. However, when only applying these manufacturing processes are applied, the performance, particularly the balance between the mechanical properties and the electrical conductivity, of the composite materials will still be relatively low. Therefore, the pre-mixing process seems to be an alternative that aids in enhancing both the mechanical properties and the electrical conductivity of the composite materials [2]. Researchers typically used a mechanical mixer to disperse fibres randomly in a polymer matrix before subjecting the materials to the manufacturing process [3]. However, no study has explained the consequence of the pre-mixing process on the electrical conductivity of composite materials. The extrusion process is a pre-mixing process that allows the fibres to be dispersed and distributed randomly in the polymer matrix. Thus, the melt mixing process using a twin screw extruder is a good alternative for the development of highly conductive polymer composite materials because this twin-screw extruder can withstand several processes, including continuous blending, premixing, and melting [4]. Moreover, pre-mixing by a twin screw extruder is an effective way of inducing the alignment of the fibre orientation in the direction of the extrusion, which will later lead to an enhancement the electrical conductivity of the polymer composite materials [5]. Methods for inducing the fibre orientation characteristics, such as the shear rate, extrusion geometry and fibre aspect ratio, are limited [5-6]. The selections of fibres used in the composite materials is crucial because long fibres offer better orientation than short fibres. During the extrusion process, a low shear rate is transferred to the long fibres, thereby maximizing the angle of orientation during extrusion process [5-6]. A high shear rate reportedly offers better fibre orientation in the polymer matrix [5]. However, at a high shear rate, the fibres experience additional rupture, which will therefore shorten the length of the fibres [7]. Considerable attention to the proper selection of the CPC materials used in the extrusion process will aid in enhancing the overall performance of the CPC [8]. Further, the appropriate die geometry, that permits the fibres to orientate accordingly, is extremely important during the extrusion process because dies play a role in shaping the molten composite exiting the extruder into the desired cross sections. In 1995, Hine reported that converging dies offer better fibre orientation compared to diverging dies when using glass fibre-reinforced polypropylene is used [9]. However, studies on the effects of fibre orientation on the electrical conductivity using the pre-mixing process are limited. Diverging dies result in elongation deformations, which allow the fibres to be randomly orientated over all the die thickness values.

Thus, converging dies are more promising for orientating the alignment of the fibres in the direction of the extrusion as fibres tend to align accordingly while passing through the narrow regions. However, high fibre loadings are needed to produce highly conductive polymer composite materials. These will later cause difficulties with regard to the wetting of the fibre particles by polymer resin, thereby leading to complications such as die blockage, during compounding using a twin screw extruder [10]. Thus, sufficient fibre loadings are important to balance between the overall performances with the capability of mixing the composite materials.

CPC materials exhibit many interesting features due to their good mechanical properties, electrical conductivity, and variation in resistivity variation with thermal and chemical stress. Moreover, the morphology and material structure of CPC materials produce an excellent conductivity pathway, which is important in producing the highly conductive materials required in bipolar plates in fuel cell applications [11]. The electrical conductivity obtained by experimental works can be verified using a conductivity model. In 1993, a conductivity model was developed based on experimental data using various materials and processing methods. However, none of the semi-empirical models are able to predict the electrical conductivity of composite materials accurately due to limitations in every developed model. Recent research showed that the GEM model has the potential to predict the electrical conductivity of composite materials. However, this model has its limitations because it predicts the electrical conductivity based on the composition of the materials and the individual material conductivity of the materials. Thus, the current research suggested the use of the modified fibre contact model by Weber and Kamal, because this model considers the fibre orientation, contact diameter between the fibres, volume fraction, and fibre aspect ratio to predict the electrical conductivity of composite materials [12]. A modified FCM model appears to have the potential to predict the electrical conductivity because more parameters are considered in the semi-empirical equations. However, this model has difficulties when it comes to defining the angle of orientation and the contact diameter between the fibres because the measurement of these parameters requires either a direct or indirect method [13]. This work was aimed at studying the effects of the die configuration on the electrical conductivity of polypropylene-reinforced milled carbon fibre, and to predict the electrical conductivity using a suitable semi-empirical model.

## 2.0 METHODOLOGY

Milled carbon fibre (MCF), with a density of 1.75 g/cm$^3$, diameter of 9 μm, and length of 300 μm, was obtained from ShenZhenYataida High-Tech. Co., Ltd. MCF has the capability to enhance the electrical conductivity at low fibre loadings, disperses evenly in a polymer matrix, and can produce highly conductive polymer composites [14]. Extrusion-grade polypropylene (PP) in powder form, with an average size of 90 μm, density of 910 kg/m$^3$, and melting index of 10 g/10 min at 160 $^o$C (Titan 600), was supplied by Goonvean Fibres Ltd. PP powder was chosen due to its impressive properties, such as good mechanical strength, low density, electrical resistance, and good permeability by gases [15]. Details of the material properties are shown in Table 1. The MCF/PP composite was physically pre-mixed in a small container at 1200 rpm for 3-4 minutes using a mechanical mixer (model RM 20-KIKA-WERK) at room temperature before the compound was melted in a Thermo Haake TSE twin screw extruder. During the extrusion process, the twin screw extruder was set at a temperature of 230 $^o$C and a rotational speed of 50 rpm for 30 minutes of extrusion time. Two different die geometries, namely sheet dies with a thickness values of 3 mm and 5 mm, and a rod die with a diameter of 5 mm, were used in this study. The in-plane electrical conductivity was measured via the four-point probe technique using a Jandel four-point probe and an RM3 test unit [3]. Meanwhile, the morphological structure of the composite material was observed using a Zeiss scanning electron microscope (SEM). Moreover, the orientation angle of the fibre was measured manually using the SEM image obtained. Then, the measured orientation angle measured was used to validate the electrical conductivity of the polymer composite material by adapting both the GEM and modified FCM models. The good correlation between the predicted model and the experimental data was studied.

Table 1: Material properties

| Material | Density (g/cm$^3$) | Size (μm) |
|---|---|---|
| Polypropylene | 0.91 | 90 |
| Milled carbon fibre (MCF) | 1.75 | 300 |

Electrical conductivity models are semi-empirical models that are useful for predicting the electrical conductivity of composite materials without requiring numerous experiments. Since the 1990s, various models have been introduced to predict either the mechanical properties or the electrical conductivity of the materials [12]. Even though the electrical conductivity models have been used in the past, current studies show that none of the models are able to predict the electrical conductivity accurately due to experimental constraints. Studies have shown that the GEM model was commonly used by the researchers due to its capability to effectively predict the electrical conductivity of single and multiple fibres [16]. However, the latest studies have reported that the GEM model is inadequate for predicting the electrical conductivity as the model only considers the material compositions and electrical conductivity individually. In addition, the GEM model also has a broad exponential value ($t$-value), which makes it difficult to precisely predict the electrical conductivity. The GEM model of multiple fibres can be found elsewhere [3]. Thus, studies have reported that the modified FCM model is preferable as this model considers more parameters, including fibre contact, fibre size and aspect ratio, angle of orientation, and volume fraction, than the GEM model, as discussed by other researchers [12-16]. Maximizing the parameters considered during the experiment will helped to minimize the gap between the predicted value and the experimental data.

## 3.0 RESULTS AND DISCUSSION

Figure 1 shows the XRD pattern of the extruded MCF/PP composite material (C and $C_3H_6$), which was mainly composed of graphite particles and polypropylene structures. This verified that the extruded MCF/PP was a pure composite, without being contaminated with other conductive elements. Moreover, from $23^0$ to $30^0$ the MCF had a wider and higher intensity compared with polypropylene from $16^0$ to $19^0$, as shown in Figure 1. This finding showed that the electrical conductivity produced using the extrusion process which was considered to be higher compared to the G/PP composite produced using the compression moulding process [17-18]. Moreover, the electrical conductivity produced by the MCF/PP composite was significant because the material needed to undergo a manufacturing process to maximize the electrical conductivity that was produced [19].

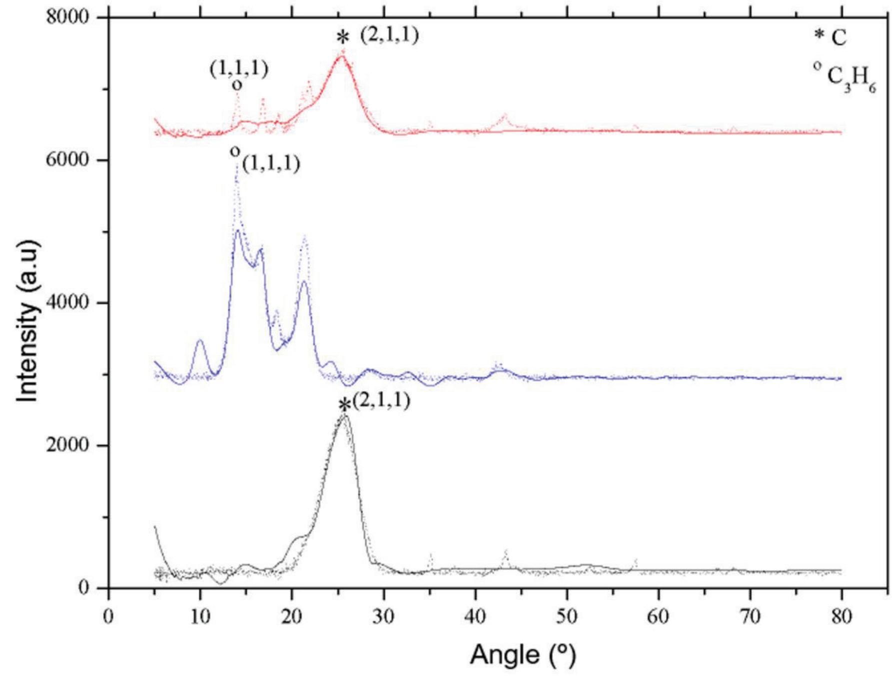


Figure 1: XRD analysis of extruded raw MCF, raw PP and MCF/PP composite

The electrical conductivity of the extruded MCF/PP composite using three different dies (e.g., rod and sheet dies), was investigated, as shown in Figure 2. The highest electrical conductivity of 3.7 S/cm was obtained from the use of the rod dies because the fibres tended to orientate better in the converging dies compared to the sheet dies [9, 20]. Converging dies offer better fibre orientation than diverging dies, thereby improving the electrical conductivity of the composite materials. In addition, the lowest shear rate from the rod dies aided in minimizing fibre rupture and in improving the fibre orientation [7]. However, previous studies reported that a high shear rate not only helps in orientating the fibre in the desired direction, but also maximizes fibre rupture, thus causing a deterioration in the electrical conductivity and mechanical properties of composite materials [12]. This could be clearly observed in Figure 2, where the electrical conductivity obtained was the lowest at the highest shear rate.

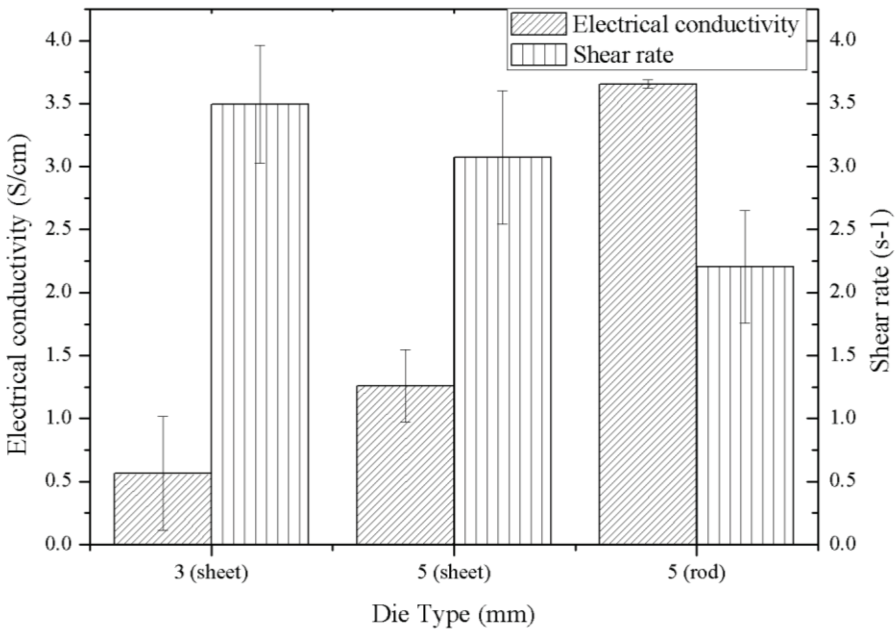

Figure 2: Electrical conductivity and shear rate of MCF/PP composites

An appropriate rotational speed is crucial to minimizing fibre rupture, even though a high shear rate enhances the orientation of the fibres. Thus, although the rod dies experienced the lowest shear rate and the rupturing of a few fibres, the electrical conductivity obtained was the highest because more fibres were extruded, leading to a dense and compact matrix. During the premixing process, it was reported that the longer fibres provided a better conductivity network compared to the short fibres [2, 6]. Moreover, the exposed fibres enhanced the overall performance of the composite materials due to the narrow orifice of the rod die, as shown in Figure 3(f). A low electrical conductivity was obtained using the sheet dies was because more fibres remained to orientate randomly in the polymer matrix. This observation was also been reported by other studies using diverging dies [9]. The rod dies offered the highest electrical conductivity compared to the sheet dies because carbon fibre has anisotropic electrical properties, which resulted in the production of a high electrical conductivity produced along the basal plane as the fibres were orientated in the direction of the extrusion [2]. Meanwhile, the sheet dies with a thickness of 5mm had a higher electrical conductivity compared to the 3 mm thick sheet dies because the reduced thickness enhanced the shear rate, thereby increasing the rupture of the fibres [7].

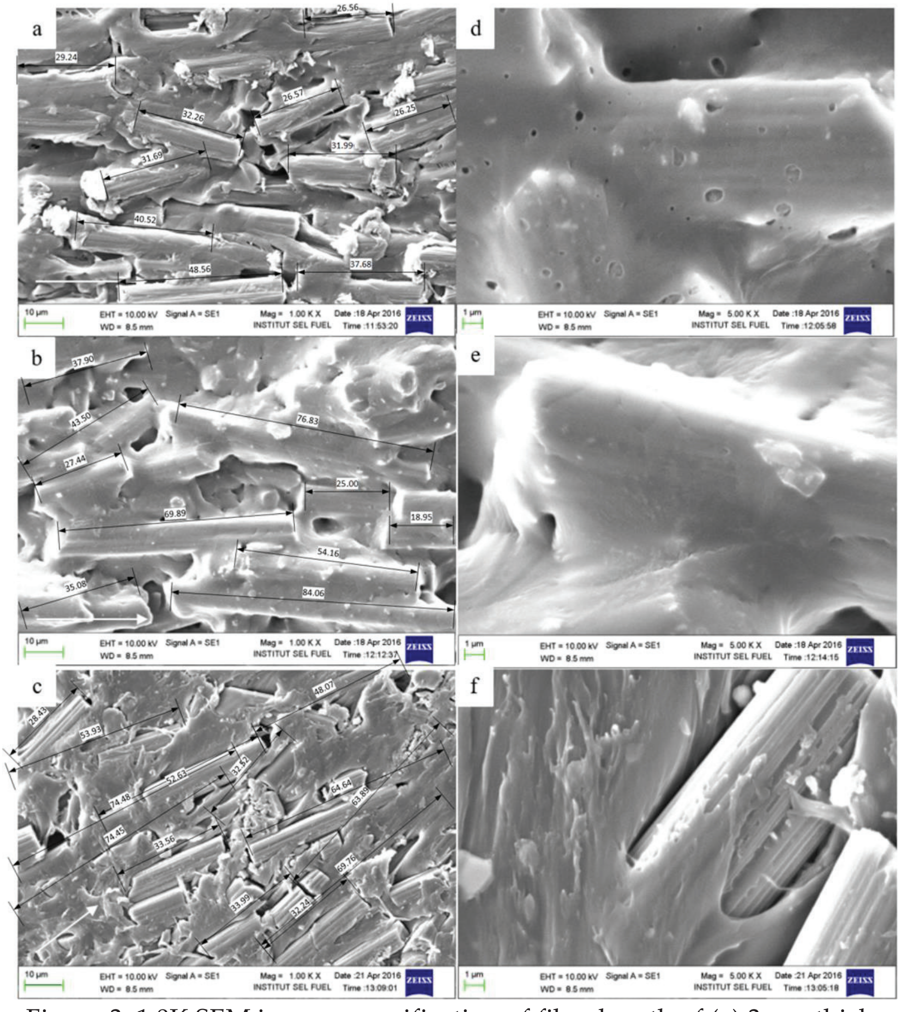


Figure 3: 1.0K SEM image magnification of fibre length of (a) 3mm thick sheet die, (b) 5mm thick sheet die and (c) 5mm wide of rod die while 5.0K SEM image magnification of (d) 3mm thick sheet die, (e) 5mm thick sheet die and (f) 5mm wide rod die

Figures 3 (a), (b), and (c) show the rod dies with the longest fibre length of 55 μm compared to the 3 mm and 5 mm thick sheet dies with lengths of 28 and 42 μm, respectively. The low electrical conductivity of the sheet dies was due to the transfer of a higher shear rate to the fibres, thereby causing fibre rupture. Meanwhile, the long rod die behaved as a converging die compared to the sheet die, which behaved as a diverging die. Hine et al. reported that the converging dies orientate fibres evenly in a polymer matrix [9]. In particular, as shown in Figures 3(d) and Figures 3(e), the polymer was fully coated and bound by the fibres, thereby minimizing the electrical conductivity of the composite materials compared to the fibres that were extruded using the rod dies, as in Figure 3(f). Moreover, the electrical conductivity obtained using the 5 mm thick sheet die was higher compared that of the 3 mm thick sheet die. This finding indicated that proper binding between the thermoplastic polymer and fibres aided in enhancing the overall electrical conductivity performance, as shown in the 5 mm thick sheet dies. Studies have reported that the thermoplastic materials experience no curing reactions, leading to the absence of gas porosity. Improper binding of the polymer matrix could be observed in Figure 3(d), where the gas porosity existed. Although the porosity is critical for highly conductive polymer composite applications, it is negligible for the pre-mixing process.

To validate the electrical conductivity obtained from the experiment, the modified FCM and GEM models were adapted into this study as shown in Figure 4. The GEM model was considered in this study as this model is currently being used by researchers to predict the electrical conductivity of composite materials [3]. However, this model was unable to predict the electrical conductivity for all the die configurations as the material compositions used in this study were identical. Thus, an electrical conductivity of 4.49 S/cm was predicted for all the dies used. These limitations in predicting the electrical conductivity were due to a lack of parameters, such as the fibre orientation, fibre aspect ratio, and contact length between fibres [10]. Thus, the rod dies offered better fibre dispersion and orientation compared to the sheet dies because the converging dies allowed the fibres to orientate as the die orifice was narrowed. This clearly showed that the fibre orientations are crucial in predicting the electrical conductivity of composite materials. The fibre orientations were measured directly using the SEM analysis [6, 21].

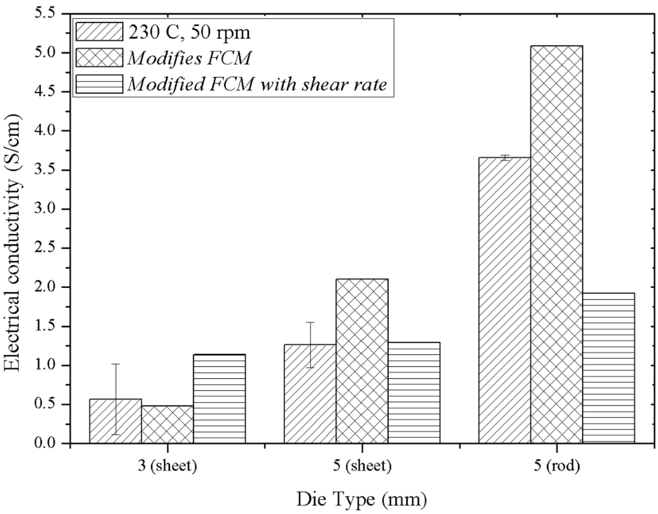


Figure 4: Electrical conductivity of extruded MCF/PP composites and electrical conductivity predicted using modified FCM model

The angle of orientation was adapted into the current modified FCM model to predict the electrical conductivity of composite materials [6]. This model considered more parameters, including the fibre-to-aspect ratio, fibre orientation, and fibre composition, compared to the GEM model, thereby producing a good correlation between the experimental data and the predicted model, as shown in Figure 4 (MFCM), with an r-square of 0.98. However, as shown in Figure 4, the predicted modified FCM model, indicated as the MFCM, had quite different experimental data compared with the modified FCM model, indicated as the MFCM with shear rate, which considered the shear rate and rotational speed as the additional parameters in the model. By considering more parameters in the model equations, the obtained predicted values that were obtained came closer to experimental data [12]. This model was altered by considering the shear rate and rotational speed of the extrusion process, as described in Equation (1):

$$\sigma_c = \sigma_m + [\frac{4}{\pi}(\frac{d_c}{d})(\frac{1}{d})(\frac{t}{s})(\cos^2\theta)(\phi_{overall}\sigma_f X)] \qquad (1)$$

where $\sigma_c$, $\sigma_m$ and $\sigma_f$ are the electrical conductivities of the composite material, matrix and fibres, respectively; $X$ is the contact number of the fibres; $d$ is the fibre diameter; $d_c$ is the fibre contact diameter; $l$ is the fibre length; $\phi$ is the volume fraction; $\theta$ is the fibre orientation angle; $t$ is the rotational speed; and $s$ is the shear rate of the composite material.

The modified FCM model showed an excellent correlation between the experimental data and the predicted model, with an R-square value of 0.99, as shown in Figure 4 (MFCM with shear rate). The fibre contact diameter $d_c$ was assumed in this model because the gap between the fibres was not measured using the SEM analysis. Moreover, the fibre contacts were assumed to be end-to-end fibre contacts instead of body-to-end and body-to-body fibre contacts [6]. Fibre contacts are critical in predicting the electrical conductivity of the composite materials because better conductivity pathways are produced, while the diameter of the contact between the fibres is reduced [12]. Thus, the diameter of the fibre contacts was assumed to be within the range of $10^{-6}$ cm to $10^{-8}$ cm, depending on the angle of the fibre orientation. As the fibres were orientated $90^0$ to the extrusion direction, the fibre contact was assumed to be $10^{-8}$cm. Meanwhile, the constant rotational speed of the extrusion process was adapted in the equations for this model because a rotational speed of 50 rpm was the optimum speed to induce the fibre orientations within the polymer matrix [5]. The shear rate was calculated based on the rotational speed of the twin screw extruder. Both parameters are important as they are considered to be the main parameters during the extrusion process. Meanwhile, the constant value, $X$ was assumed to be 2.85, based on the mathematical relationship between the numbers of fibre contacts. The details of the relationships are discussed elsewhere [6].

Unlike the rod dies, the sheet dies exhibited more randomly orientated fibres due to the high shear rate, which maximized the fibre ruptures, as shown in Figures 3(a), (b) and (c). Researchers have explained that the shorter fibres in the sheet dies indicate that the fibres are orientated randomly within the polymer matrix due to the low viscosity and shear rate [5]. Moreover, they reported that a prolonged cooling time during the extrusion process also affects the fibre orientation. Meanwhile, a study reported that the mechanical properties and the overall performance of the composite materials starts to deteriorate as the orientation angle widens (below $90^0$). Thus, the average fibre orientations were assumed to be within $50^0$ to $60^0$ for the sheet dies and $85^0$ to $90^0$ for the rod dies. The contradicting angle of orientation between the rod dies and the sheet dies was related to the shear rate produced, as low shear rate results in long fibres. Studies have reported that the fibre orientation depends on the aspect ratio of the fibres used, as long fibres rather than short fibres, produced a large angle of orientation [20]. Figure 3 shows that the fibre orientation and dispersion were governed by various factors, including the die geometry and fibre aspect ratio [5]. Meanwhile, the shear rate and rotational speed were considered in this model as both are the main parameters that govern the fibre orientation [10]. This model is also applicable for composites that are subjected to manufacturing processes, such as injection moulding and compression moulding.

## 4.0 CONCLUSION

The electrical conductivity of an extruded MCF/PP composite using a rod die geometry with a diameter of 5 mm was superior to that of sheet dies with a thickness of 3 mm and 5 mm because of the better fibre orientation in the converging dies. The electrical conductivities of the rod die and the sheet die with a thickness of 5 mm were 3.7 and 1.3 S/cm, respectively. The results obtained were validated using an electrical conductivity model, i.e., a modified FCM that took into consideration the shear rate and rotational speed (MFCM with shear rate). There was excellent correlation with the experimental data compared to the GEM model with an electrical conductivity of 4.49 S/cm and the modified FCM model. The rod dies enhanced the electrical conductivity of the composite material due to the compactness of the fibre and their exposure during the extrusion process as the lowest shear rate minimized the fibre ruptures. Further research is needed to explore an improved method for measuring the orientation angle and diameter of the fibre contact accurately to establish the modified FCM model.

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